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MEMORANDUM REPORT ARBRL-MR-03219

RELATIONSHIP OF  
COMBUSTION CHARACTERISTICS AND  
PHYSICAL PROPERTIES OF BLACK POWDER

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November 1982



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND  
BALLISTIC RESEARCH LABORATORY  
ABERDEEN PROVING GROUND, MARYLAND

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20. (continued) evidence of *combustion*-induced deconsolidation.

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## I. INTRODUCTION

Black powder exhibits a number of combustion properties which makes it very useful as an igniter component in a propelling charge. It has a relatively high ignition temperature which makes it safer, under some conditions, from accidental ignition when compared with single, double, and triple base propellants. However, once ignited, black powder burns very vigorously, even under sub-atmospheric conditions, exhibiting rapid flame-spread properties. On the other hand, other propellants burn slowly at low pressure and, in some instances, will only fizz burn. Since ignition takes place initially at one atmosphere, where black powder excels, it has been used extensively in ignition systems. In fact, due to the shortage of domestic suppliers, the Army Corps of Engineers and ICI Americas, Inc., have constructed a black powder production facility at the Indiana Army Ammunition Plant (IAAP) in Charlestown, Indiana<sup>1,2</sup>. In spite of many centuries of use, the fundamental combustion and flamespread characteristics of black powder are not well understood and, in some cases, not even characterized. This lack of knowledge has led to vague and uncertain specifications for the manufacture of black powder<sup>3,4</sup>. In order to address this specification problem, black powder lots were manufactured in which specific deviations to the conventional processes were introduced. These lots were tested in igniter systems<sup>5</sup> and in a flamespread measuring device<sup>6</sup>. Differences in results were noted between the studies which were not clearly resolved.

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<sup>1</sup>J. R. Plessinger and L. W. Braniff, "Development of Improved Process for the Manufacture of Black Powder," RCS AMURE-109, Project No. 5714170, Olin Corp., Indiana Army Ammunition Plant, Charlestown, IN, December 1971.

<sup>2</sup>H. E. Carlton, B. B. Bohrer, and H. Nack, "Advisory Services on Conceptual Design and Development of New and Improved Processes for the Manufacture of Black Powder," Final Report, Battelle Memorial Institute, Columbus, OH, October 1970.

<sup>3</sup>J. C. Allen, "The Adequacy of Military Specification MIL-P-223B to Assure Functionally Reliable Black Powder," ASRSD -QA-A-P-60, Picatinny Arsenal, Dover, NJ, June 1974.

<sup>4</sup>J. C. Allen, "Concept Scope of Work for MM&TE Project 5764303 Acceptance of Continuously Produced Black Powder," SARPA-QA-X-010, Picatinny Arsenal, Dover, NJ, November 1975.

<sup>5</sup>K. J. White, H. E. Holmes, and J. R. Kelso, "Effect of Black Powder Combustion on High and Low Pressure Igniter Systems," 16th JANNAF Combustion Meeting, CPIA Publication 308, Vol. I., pp. 477-497, December 1979.

<sup>6</sup>N. A. Messina, L. S. Ingram, and M. Summerfield, "Black Powder Quality Assurance Flamespread Tester," PCRL-TR-78-101, Princeton Combustion Research Laboratories, Princeton, NJ, December 1978.

In an earlier investigation, an attempt at modifying the manufacturing process of black powder<sup>7</sup> was stymied by inexplicably large differences in burn rates between lots as determined by closed chamber studies.

This report will examine the anomalies outlined above and will attempt to show the importance of physical and mechanical properties on the performance of black powder. Grain break-up is believed to be the cause of a number of reported phenomena and evidence for this will be presented. Since the manufacturing process dictates the physical properties, a brief description of the process will be given here. Such a discussion should help clarify the rationale for the experiments conducted in this study.

Black powder is a mixture of 75% potassium nitrate, 15% charcoal and 10% sulfur. These ingredients are incorporated by several techniques<sup>2,7,8</sup>, which are summarized. The "as-received" potassium nitrate is crushed and beaten to a fine dust. Lumps of charcoal and crystalline sulfur are ball-milled together. The charcoal-sulfur mixture is incorporated with the potassium nitrate in a wheel mill with a few percent water added. (The Norwegian process, adapted by IAAP, uses a jet mill instead of a wheel mill.) The mixture from the wheel mill is then broken up and placed in a press where it is compacted into large "cakes" of proper density. The "cake" is then broken up and passed through a corning mill where it is fractured into the desired granulation sizes. These grains are irregular in shape and range in effective diameter from 0.05 mm up to several mm. The grains are then tumble-dried in large barrels where graphite powder is added during the drying process. The graphite-coated black powder granules are removed and screened into specific granulation sizes and packaged. Details differ from manufacturer to manufacturer<sup>2</sup>, but it is clear that numerous steps in the process involve procedures that affect the physical properties of the product. Moreover, pressure, moisture, tumbling time, milling time, and other parameters are largely determined through experience and operator intuition which can vary from lot-to-lot.

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<sup>7</sup>Propeller Chemical Division, "A Study of Modernized Techniques for the Manufacture of Black Powder," DAI-23-072-501-ORD-(P)-43, Edwardsville, IL, January 1960. (AD 232645)

<sup>8</sup>T. Urbanski, *Chemistry and Technology of Explosives*, Vol. III, translation by M. Jurecki, edited by S. Laverton, Pergamon Press, NY, NY, 1967, Volume 3, Chapter III.



Various investigators<sup>1,7,8,9,10,11,12,13</sup> have recognized the importance of these physical as well as chemical properties, and they have tried to measure their effect on ballistic performance. The work reported here attempts to extend these investigations such that manufacturing procedures and variables can be identified and controlled to give predictable and reproducible performance of black powder. In addition, it should be noted that the British government has initiated a similar program to investigate the properties of black powder<sup>14</sup>, since they have observed ballistic problems in several weapons systems which are directly attributable to inadequate performance of black powder.

## II. EVIDENCE FOR GRAIN BREAK-UP

This section will present evidence from several different tests and experiments which together suggest that black powder, under certain conditions, may break up while burning. This phenomenon would lead to increased surface area during combustion with a concomitant increase in gas generation rate. The grain break-up phenomenon also suggests that the mechanical properties of black powder should be considered both in the design of igniter components and also in the manufacturing of black powder. Well-controlled combustion properties might not be sufficient if the igniter device promotes grain break-up of the black powder to the extent of dominating the combustion process. Such effects could be amplified if the lot-to-lot strength varied due to manufacturing conditions.

The evidence to be presented includes:

a. flamespread measurements under different conditions gave results that could be explained by grain break-up,

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<sup>9</sup>R. A. Sasse', "The Influence of Physical Properties on Black Powder Combustion," ARBRL-TR-02308, Ballistic Research Laboratory, USAARRADCOM, Aberdeen Proving Ground, MD, March 1981.

<sup>10</sup>J. E. Rose, "Investigation on Black Powder and Charcoal," IHTR 433, Naval Ordnance Station, Indian Head, MD, September 1975.

<sup>11</sup>Propellex Chemical Division, "Engineering Studies to Develop an Improved Method for the Manufacture of Black Powder," DAI-23-072-501-ORD-(P)-43, Edwardsville, IL, July 1961. (AD 259642)

<sup>12</sup>W. Hintze, "Einfluss des Kohlenstoffgehaltes der Holzkohle auf die Schwarapulvereigenschaften," *Explosivstoff* 2, pp. 41-48, 1968.

<sup>13</sup>W. Hintze, "Einfluss der Feuchtigkeit auf die Qualitaetseigenschaften des Schwarapulvers," *Nobel Hefte* 2, pp. 14-21, 1972.

<sup>14</sup>E. McAuliffe, and H. Marsh, "The Use of Charcoal in Blackpowder. A Study of Structure and Properties of Wood Charcoal," 3rd International Carbon Conference, Baden-Baden, GE, July 1980.

b. comparisons of closed chamber measurements to strand burner results show very different burning rates,

c. closed chamber results show large variations from sample to sample depending on how the black powder is prepared.

#### A. Various Methods Used for Flamespread Measurements

Flamespread velocity measurements were made for the deviant lots of Class 1 black powder. Class 1 black powder is an irregularly shaped grain in which 3% (max.) is retained on United States Standard screen size number 4 (screen opening, 4.76mm) and 5% (max.) passes through screen size number 8 (screen opening, 2.38mm.) Flamespread velocity measurements for the deviant lots of Class 1 black powder were carried out at three laboratories using different techniques<sup>5,6</sup>. Princeton Combustion Research Laboratories (PCRL)<sup>6</sup> used a metal tube with a series of transverse slot-holes cut at intervals down the length of the tube. Black powder that filled the tube was ignited with a soft igniter system at one end, and the propagation velocity was measured with light detecting diodes. ARRADCOM, Large Caliber Weapons System Laboratory (LCWSL), measured relative quickness (RQ) of the lots in a closed chamber<sup>5,6</sup>. ARRADCOM, Ballistic Research Laboratory (BRL), measured the open-air flamespread velocity. BRL also employed an M28B2 primer (the primer used in the 105-mm howitzer) which consists of a metal tube with round venting holes down its length, having a similar venting area to the PCRL device. However, the M28B2 primer uses a brisant M61 initiator to ignite the black powder. Tests with inert (boiling chips) simulants have shown that the M61 breaks up and compacts the boiling chips. Propagation velocity for the deviant lots was measured using pressure gauges and high speed cinematography<sup>5</sup>. The results from the PCRL, M28B2 and open-air flame spread tests are summarized in Figure 1; they are normalized to Lot GOE 75-44.

Data from these tests show the same general trends, i.e., Lot 1 is slower than Lot 2 which is faster than Lot 3, etc. However, there is one major difference in comparing the three data sets of Figure 1. The PCRL device data show a substantially lower performance for Lots 1-10 compared with GOE 75-44. The open-air flamespread data are in between these two data sets. Closed chamber results (not shown in Figure 1) are similar to the open-air flamespread results. As will be discussed in Section III, there is no evidence for grain break-up or deconsolidation during combustion of black powder when a hot wire is used as an igniter. Consequently, the open-air flamespread results could reflect ignition and combustion properties of black powder but not its mechanical properties. However, the PCRL device results and, especially the M28B2 primer measurements, may be affected by the mechanical properties of the black powder due to both the brisant initiator (M61) and to the confinement of the powder by the tube. For example, the propagation velocity (propagation velocity is the velocity of the flame front in traveling down the primer tube) of the M28B2 may be dominated by the mechanical properties of the black powder due to the sharp pressure pulse generated by the M61 initiator. The PCRL flamespread device results show grain break-up only with Lot 11 and GOE 75-44. Both of these lots are wheel-milled, Gearhart-Owen black powder using maple charcoal. Lots 1-10 were made with the jet mill and used oak charcoal<sup>9</sup>.

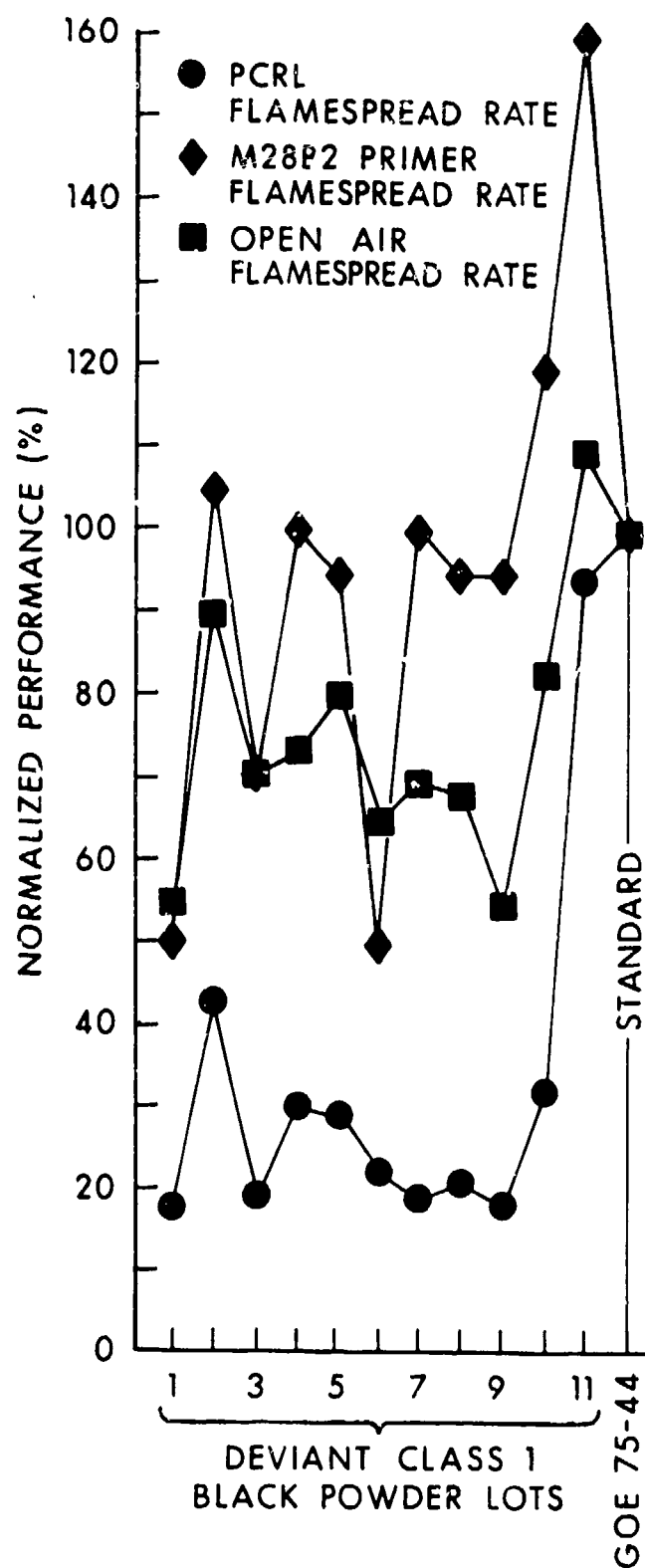


Figure 1. Normalized Flamespread Performance Data of Various Class 1 Black Powders

These remarks are speculative and can only be proven by repeating the M28B2 primer tests using a very "soft" igniter such as a hot wire; measurement of the mechanical properties of the different lots may show significant differences. These tests have not yet been carried out.

#### B. Closed Chamber and Strand Burner Studies

Price and Juhasz<sup>15</sup> have calculated burn rates from closed chamber combustion studies of Class 1 black powder. Tests were carried out with a loading density of 0.18 g/cm<sup>3</sup> and the chamber was equipped with a brisant M52 igniter. An average spherical grain geometry (3.8-mm diameter) was assumed for the analysis. A burn rate expression derived from the Price-Juhasz data is:

$$r_p = 12.2 \, p^{0.56} \qquad 2.5 \text{ MPa} < P < 25 \text{ MPa} \quad (1)$$

where P is pressure in MPa and r is burn rate in mm/s.

Strand burn rate measurements were carried out by Russian researchers<sup>16,17,18</sup> on pellets of black powder burned in a pre-pressurized chamber. The loading density was such that the pressure rise during combustion was very small. Burn rates were deduced from streak camera and ion gauge measurements. Other measurements were made with a stack of pellets differing in diameter. Pressure discontinuities located transitions from one diameter to another. The sides of the pellets were chemically inhibited. The strand burner results are plotted in Figure 2. Both Williams<sup>19</sup> and Rose<sup>20</sup> have discussed these results.

The Belyaev data yield the following expression:

$$r_b = 21.2 \, p^{0.188} \qquad 0.5 \text{ MPa} < P < 300 \text{ MPa} \quad (2)$$

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<sup>15</sup>C. F. Price, Naval Weapons Center, China Lake, CA; A. A. Juhasz, Ballistic Research Laboratory, APG, MD; private communication.

<sup>16</sup>A. F. Belyaev and S. F. Maznev, "Dependence of Burning Rate of Smoke-Forming Powder on Pressure," *Dokl. Akad. Nauk SSSR* **131**, pp. 837-889, 1960.

<sup>17</sup>A. P. Glazkova and I. A. Tereshkin, "Relation Between Pressure and Burning Velocity of Explosives," *Zhur. Fiz. Khim.* **35**, pp. 1622-1628, 1961.

<sup>18</sup>A. F. Belyaev, A. I. Korotkov, A. K. Parfenov and A. A. Sulimov, "The Burning Rate of Some Explosive Substances and Mixtures at Very High Pressures," *Zhur. Fiz. Khim.* **32**, p. 150, 1963.

<sup>19</sup>F. Williams, "The Role of Black Powder in Propelling Charges," TR 4770, Picatinny Arsenal, Dover, NJ, May 1975.

<sup>20</sup>J. L. Rose, and A. P. Hardt, "Black Powder - A Modern Commentary - 1979," *Proceedings of the 10th Symposium of Explosives and Pyrotechnics*, Franklin Research Center, Philadelphia, PA, February 1979.

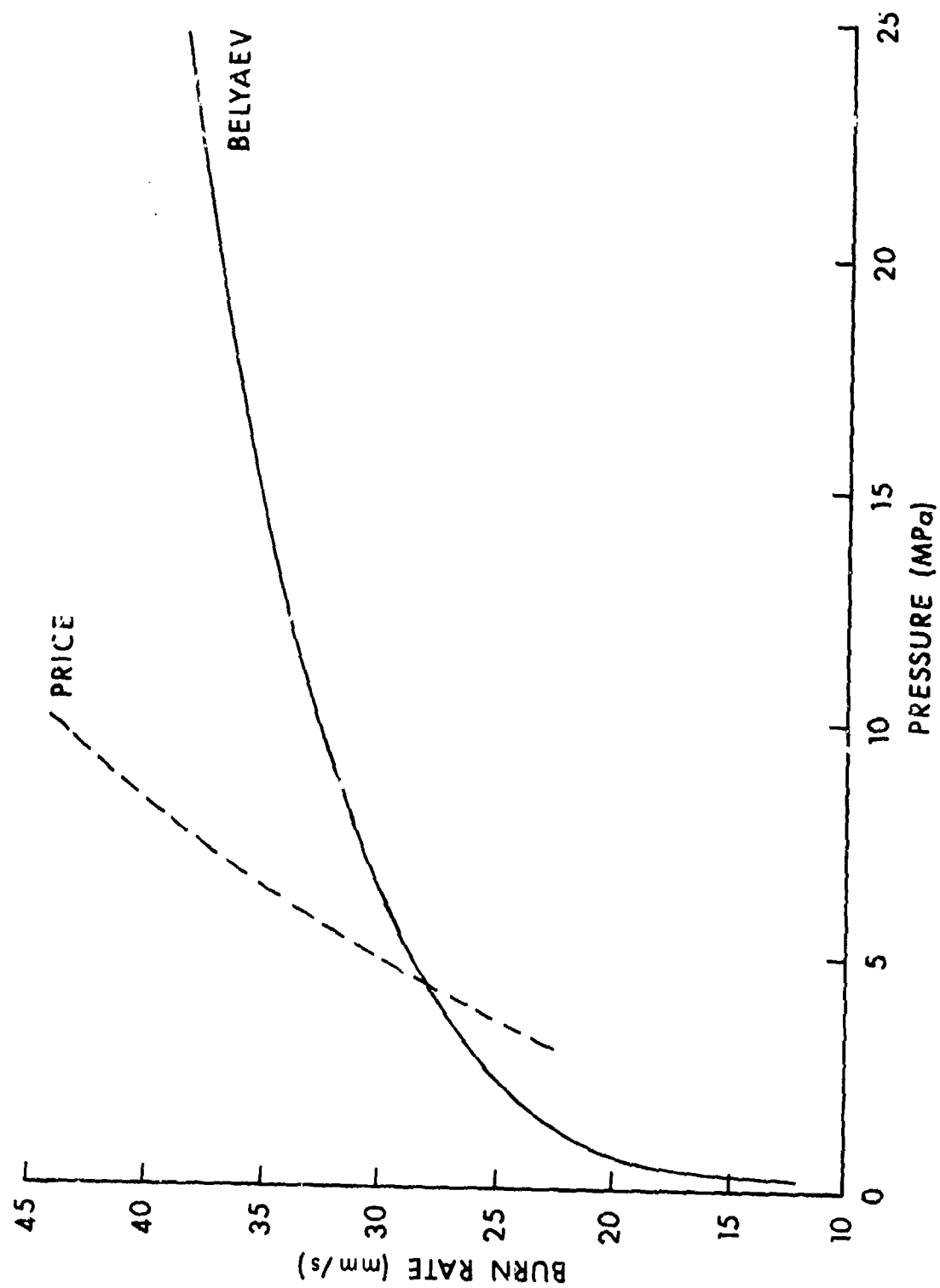


Figure 2. Burn Rate of Black Powder as a Function of Pressure for Closed Chamber (Price) and Strand Burner (Belyaev) Experiments.

In independent tests with small black powder rocket motors, Barber<sup>21</sup>, et al, have found the burn rate of black powder to be:

$$r_{ba} = 20.1 p^{0.133}$$

$$0.5 \text{ MPa} < P < 2.7 \text{ MPa} \quad (3)$$

The Barber results are remarkably similar to those of Belyaev considering the different techniques and materials employed. However, there is a major discrepancy between the Belyaev and Price-Juhasz burn rate exponent. The igniter used in the closed bomb experiments of Price-Juhasz was a very vigorous M52. This could cause fracturing of the black powder grains during combustion which would lead to a higher gas generation rate as a function of pressure. It is known that Belyaev used very high consolidation pressures (over 400 MPa) in making the black powder pellets used in the strand burner experiments. Industrially made black powder, such as that used by Price-Juhasz, is pressed into "cakes" at approximately 20 MPa<sup>2,8</sup>. The "cakes" are then broken up into irregularly shaped grains. The large difference in pressing conditions, such as dwell time, pressure, water content and particle size, could cause differences in the mechanical properties of the final product, causing one to be more frangible than the other. Because of the different burn rate exponents reported, a series of burn rate measurements on strands of black powder was carried out in a windowed combustor. Using high speed cinematography, it was possible to measure the burn rate directly, and, at the same time, record any deconsolidation or break-up that might occur. These tests will be described in Section III.

### C. Propellex Black Powder Study

In the late 1950's, the Propellex Chemical Division, Chromalloy Corporation, Edwardsville, IL, conducted an investigation using new techniques for the manufacture of black powder<sup>7,11</sup>. Resulting powders were evaluated in a closed chamber. The results from some of these tests are shown in Figure 3. Commercial I and II are Class 1 (FG) black powder, of conventional manufacture, and PCD"B" and PCD"A" were black powders formulated by Propellex. The standard ingredients were mixed and pressed into "cakes" (25 mm in diameter and 50-75 mm in length). The cakes were broken and sieved into a Class 1-sized grains and graphite coated. PCD#6 and #7 are symmetrical, smooth pellets. Unfortunately, due to some inconsistencies in Reference 7, the actual values for  $dP/dt$  are not given; also samples PCD#1-5 are not included in Figure 3. What is important in this figure is the general shape of the curves. The irregular grains yield a monotonically increasing function for  $dP/dt$  with pressure whereas the pellets show an almost constant function after an initial rise. The shape of an individual pellet or grain is such that if all surfaces are burning, the surface area will decrease with time, as will the gas generation rate. *If the surface area is regressive, how can  $dP/dt$  be increasing with pressure?* This question can be answered if the burn rate is increasing rapidly with pressure. The burn rates given in the last section have very different pressure dependencies.

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<sup>21</sup>A. H. Barber, R. Parks, and C. Flanigan, "The Pressure and Temperature Dependent Properties of Black Powder Propellants," MIT Rocket Society J. 6, pp. 17-24, 1975.

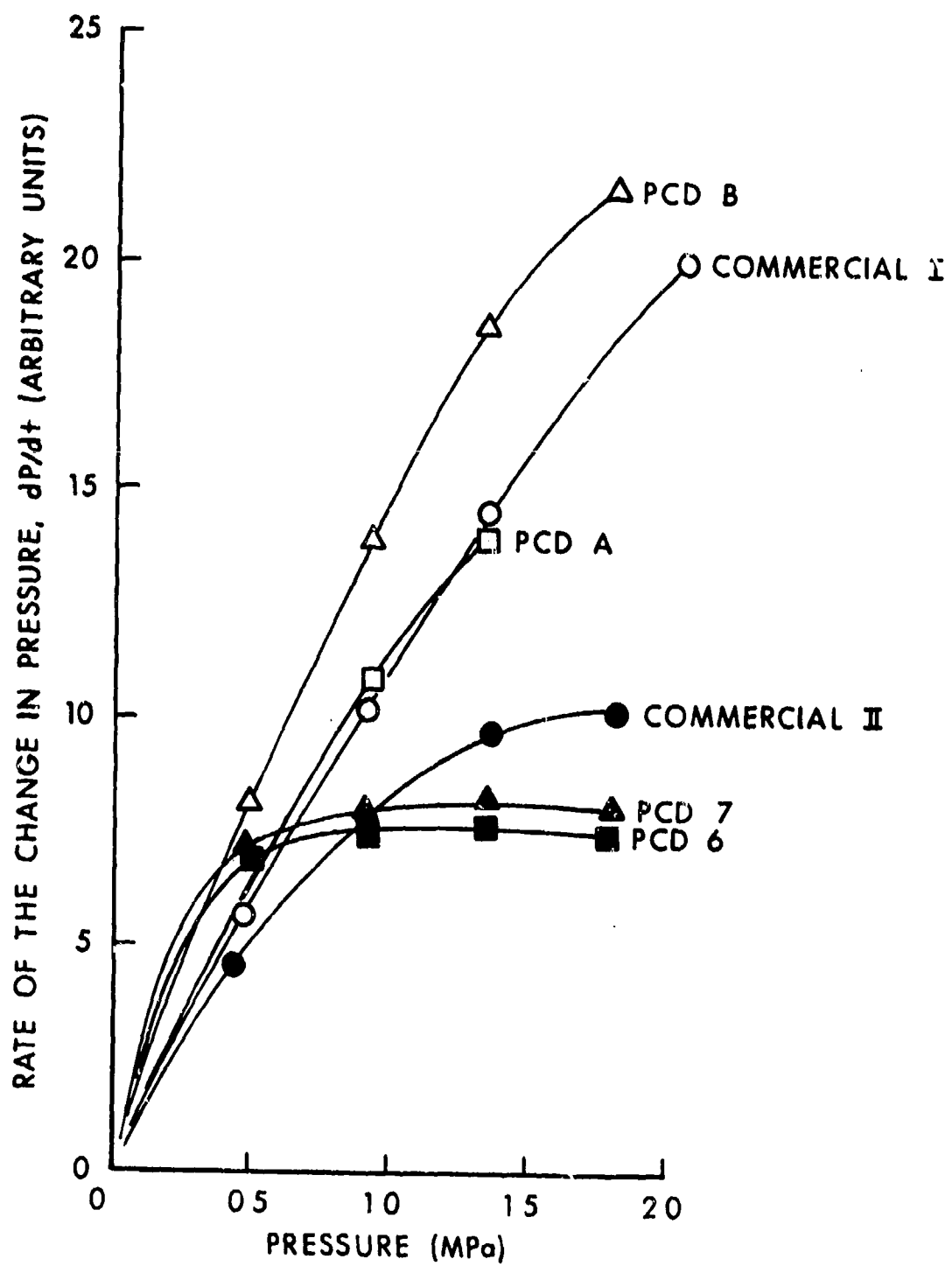


Figure 3. Closed Chamber Measurements;  $dP/dt$  vs.  $P$ .

$$r_p = 12.2 \quad p^{0.56} \quad (1)$$

$$r_b = 21.2 \quad p^{0.188} \quad (2)$$

Such differences could explain the different  $dp/dt$  data for the granules and pellets.

To show this, a closed chamber simulation computer program<sup>22</sup> was used to generate  $dp/dt$  vs.  $P$  curves using these two burn rates and standard thermochemical values for black powder reactions. The grain shape was assumed to be cylindrical (4-mm diameter and 2.4-mm length) in both cases. The results are shown in Figure 4. Comparing Figures 3 and 4, it is seen that the qualitative shape of the curves are reproduced by the model if it is assumed that  $r_p$  represents the burn rate for the black powder grains (PCB"A", PCB"B", Commercial I, II) and  $r_b$  that of the pellets (PCD #6 and #7). It should be noted here that pellets from PCD #6 and #7 were produced in a pelletizing machine with compaction pressures of 68MPa, much higher than that used in the commercial process (20 MPa), or that used in producing PCB"A" and PCB"B" (30 MPa).

*Could these results be explained by the fact that one burn rate represents the combustion of grains and the other the combustion of pellets? In a later program<sup>11</sup>, other black powder formulations were made by Propellex and they were produced in granular form. Nevertheless, the same  $dp/dt$  vs.  $P$  trends were observed as with PCD#6 and PCD#7. Consequently, the answer to the above question is probably "no".*

*Is it possible that the pellets had a greater mechanical strength since they were consolidated with a greater pressure than the granular material? To test this hypothesis, experiments were carried out in a windowed combustor to look for any evidence of combustion induced grain break-up. This will be discussed in Section III.*

The closed chamber model assumes that the entire black powder bed ignites simultaneously. *Is it possible that this is not the case and that what is being observed for the Commercial I and II powders is a "progressivity" induced by a flamespread phenomenon? This is a difficult question to answer. The pellets were observed to have smooth shiny surfaces, and in fuse development work<sup>23</sup> it has been observed that smooth surfaces inhibit flamespread. However, a slow flamespread mechanism should result in an increasing surface area with time and, consequently  $dp/dt$  should increase with pressure. This was not observed with the burning black*

<sup>22</sup>F. W. Robbins, and A. W. Horst, "Numerical Simulation of Closed Bomb Performance Based on Blake Code Thermodynamic Data," IHMR 76-259, Naval Ordnance Station, Indian Head, MD, November 1976.

<sup>23</sup>Naval Ordnance Laboratory, Ordnance Explosive Train Designers Handbook, NOLR 1111, Naval Ordnance Laboratory, White Oak, MD, April 1952.



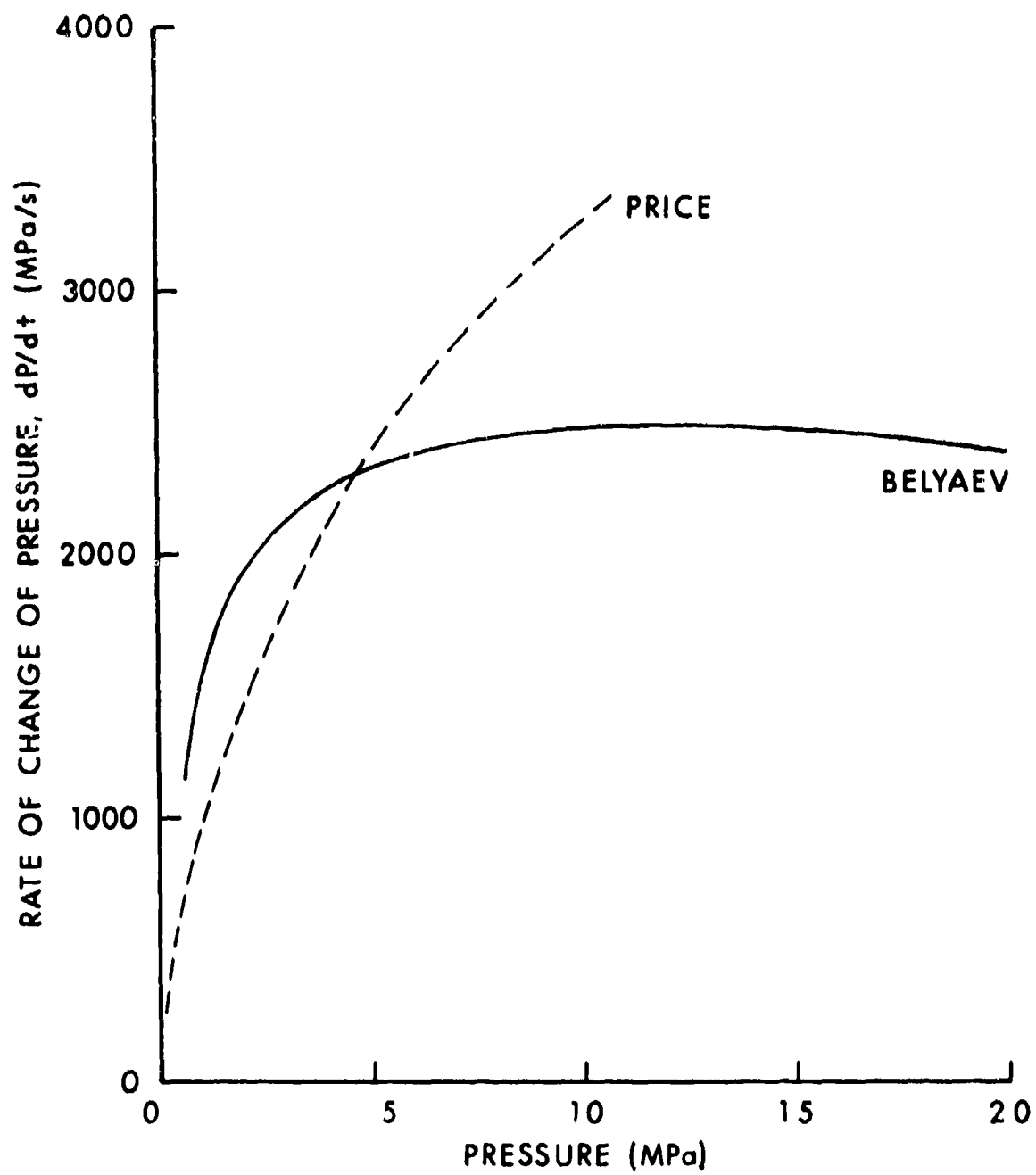


Figure 4. Closed Chamber Calculations;  $dp/dt$  vs.  $P$ .

powder pellets. The graphite coating is known to inhibit flamespread, but all samples, whether granules or pellets, had a graphite coating. However, the quality or quantity of the coating was not reported. Furthermore, the rough irregular surface of the granular black powder should enhance the flamespread velocity since the Reynolds number and heat transfer coefficient will be larger than for smooth pellets. Considering all of the above, it would seem that the pellets should exhibit a greater resistance to flamespread than the Class 1 granules, but the  $dp/dt$  vs.  $P$  data show the opposite effect. In answer to the above question, it would appear that flamespread is not an important phenomenon in explaining the results in Figure 3.

*Could the  $dp/dt$  vs.  $P$  data for the pellets be explained by heat loss to the closed chamber walls?* It was observed that the burn time for PCD#6 and #7 was almost three times longer than for the Class 1 granular powder. The greater heat loss for the slower burning pellets would decrease  $dp/dt$  at high pressures. Unfortunately, the original pressure vs. time data were not available to estimate the difference in heat loss between the granular and pellet data by examining the pressure decay portion of the curve. Without these data, the importance of heat loss cannot be assessed.

Up to this point in the discussion, a number of arguments have been advanced to explain the Propellex results. Ignoring, for the moment, the possible contribution due to slow flamespread and heat loss, the results can be explained either by the existence of two burn rates for black powder or by invoking a grain break-up or deconsolidation mechanism during combustion. There is other evidence that supports the latter argument. Propellex<sup>9</sup> manufactured two sets of samples from "batch No. 2". These were pressed into cakes, broken up and sieved into Class 1 granulation. One set had a density of  $1.65 \text{ g/cm}^3$  and a relative quickness of 136%. The other set had a density of  $1.74 \text{ g/cm}^3$  and a relative quickness of 51.5%. Grain break-up of the lower density sample could explain the high RQ.

*Could these differences be explained by the fact that Propellex powder is intrinsically different from commercial powder?* To answer this question, Propellex also pressed pellets (4-mm diameter and 2.4-mm length) using commercial FFFG (Class 6) black powder. Some firings from this material showed nearly a constant value for  $dp/dt$ , and others showed a steeply rising function. An erratic and unpredictable grain break-up could be responsible for these irreproducible results, even with the pellets. This test shows, however, that the nearly constant  $dp/dt$  phenomenon was not confined to powders made by Propellex but was also observed with commercial powders.

#### D. Naval Ordnance Station Closed Chamber Studies

Rose<sup>10</sup> has carried out a study in which closed chamber firings were conducted with a number of different lots and several granulations of black powder. He defined a specific parameter  $\phi_1$  as

$$\phi_1 = \frac{\text{time from 0.34 MPa to inflection point}}{\text{time from 0.34 MPa to peak pressure}}$$

When the value of this parameter is 0.5 the material is considered to be "neutral" burning. Larger values indicate "progressive" burning and smaller values, "regressive" burning.

In almost all cases,  $\theta_1 \approx 0.5$  for the Class 2 black powder. The value of  $\theta_1$  for the Price-Juhasz<sup>1</sup> burn rate expression is also on the order of 0.5.<sup>1</sup> However, the Belyaev burn rate expression, when applied to a closed chamber calculation, yields a value for  $\theta_1$  of 0.2. It is clear, then, that the closed chamber results of Rose are similar to those of Price-Juhasz. This again may be evidence for grain break-up, induced by the closed chamber configuration.

#### E. Graphite Coating on Black Powder

One of the final steps in the production of black powder (DuPont, Gearhart-Owen), consists of loading large drums with granulated black powder (from 200-2000 kg), and tumbling until a specified moisture level is achieved. The drum is stopped, graphite is added, and the tumbling is continued. The entire process can take from 16 to 17 hours; less time is required if steam is added. During tumbling there is a substantial rise in temperature (90°C) due to frictional heating, and a small increase in density (1.65 to 1.72 g/cm<sup>3</sup>). Consequently, this drying and graphiting process could induce some physical changes in the material. The Norwegian (Nitedal) and Italian-Swiss (Biazzi) processes employ separate stages for polishing and graphite coating<sup>2</sup>. Such samples have been evaluated by Plessinger<sup>3</sup>, who has carried out closed chamber studies on black powder with and without the graphite coating. The results are given in Table 1.

TABLE 1. EFFECT OF GRAPHITE ON BLACK POWDER

<u>Sample</u>	<u>Relative Quickness (RQ)</u>	<u>Relative Force (RF)</u>
DuPont - with graphite	100	100
DuPont - without graphite	100	99
Olin - with graphite	67	100
Olin - without graphite	113	103
Nitedal - with graphite	76	100
Nitedal - without graphite	100	99
Biazzi - with graphite	57	97
Biazzi - without graphite	85	97

Both DuPont powders listed in Table 1 went through the same tumbling process. For both graphited and nongraphited materials, the relative quickness (RQ) was the same. On the other hand, Nitedal and Biazzi "without graphite" products skipped one step in the production process, namely, the tumbling time required for graphite coating. In one case, Biazzi reduced the tumbling time of a 600 kg load by nine hours. Note that the "without graphite" powder has an RQ greater than the finished powder. If grain break-up is occurring, the graphited powder may have stronger mechanical properties due to increased tumbling time and, hence, show a smaller RQ. This is also true for the Nitedal black powder. Unfortunately, the history of the production of the Olin powder cannot be recalled; therefore, it is not clear at this time what the exact process steps were for these samples.

The results could be explained by postulating that the graphite coating induces a slower burn rate. However, measurements with a scanning electron microscope (SEM) show that graphite coating (DuPont, Gearhart-Owen sample) has a thickness of approximately 5  $\mu\text{m}$ . The influence of such a thin layer on the burn rate would not persist long into the pressure-time history, and it should have little affect on the RQ. The coating could influence the flamespread rate<sup>24</sup> in these tests and cannot be dismissed in explaining these results. However, in the light of evidence from the previous sections, mechanical properties may well explain the results in Table 1.

It would be useful to summarize the results presented in this section:

- a. the performance of a given lot of black powder depends strongly on the ballistic test device,
- b. the burn rate exponent of black powder determined in a closed chamber is three times larger than that determined from strand burning,
- c. a large difference in  $dp/dt$  was observed depending on whether the black powder was in the form of irregular grains or pellets,
- d. the graphite coating had a substantial effect on the closed chamber relative quickness.

To explain these results, it is postulated that black powder exhibits one or both of the following phenomena:

- a. two different burn rates
- b. grain break-up.

### III. WINDOWED COMBUSTOR STUDIES

The results from the previous section indicated the need for an experiment which would measure the burn rate of black powder and, at the same time, test for the possibility of grain break-up. A windowed combustion chamber and high speed cinematography offered this capability. A schematic of the experiment is shown in Figure 5. The "chimney" burner is similar to that described in Reference 25, although lower flow rates through the nozzle were used in the present investigation. The chamber was pre-pressurized at pressures from ambient up to 13 MPa. A loading density of  $2.0 \times 10^{-3} \text{ g/cm}^3$  insured minimum pressure rise during combustion. Black powder samples were molded into parallelepipeds, 4 by 5 by 20 mm; they were ignited at one end

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<sup>24</sup>M. Vielle, *Memorial Des Poudres Et Salpêtres*, Vol. 6, Chapter II, pp. 256-391, Gauthier-Villars Et Fils, Imprimeurs-Librarires, Paris, 1893.

<sup>25</sup>N. Kubota, T. J. Ohlemiller, L. H. Caveny and M. Summerfield, "The Mechanism of Super-Rate Burning of Catalyzed Double Base Propellants," Report No. AMS 1087, Dept. of Aerospace and Mechanical Sciences, Princeton University, Princeton, NJ, March 1973.

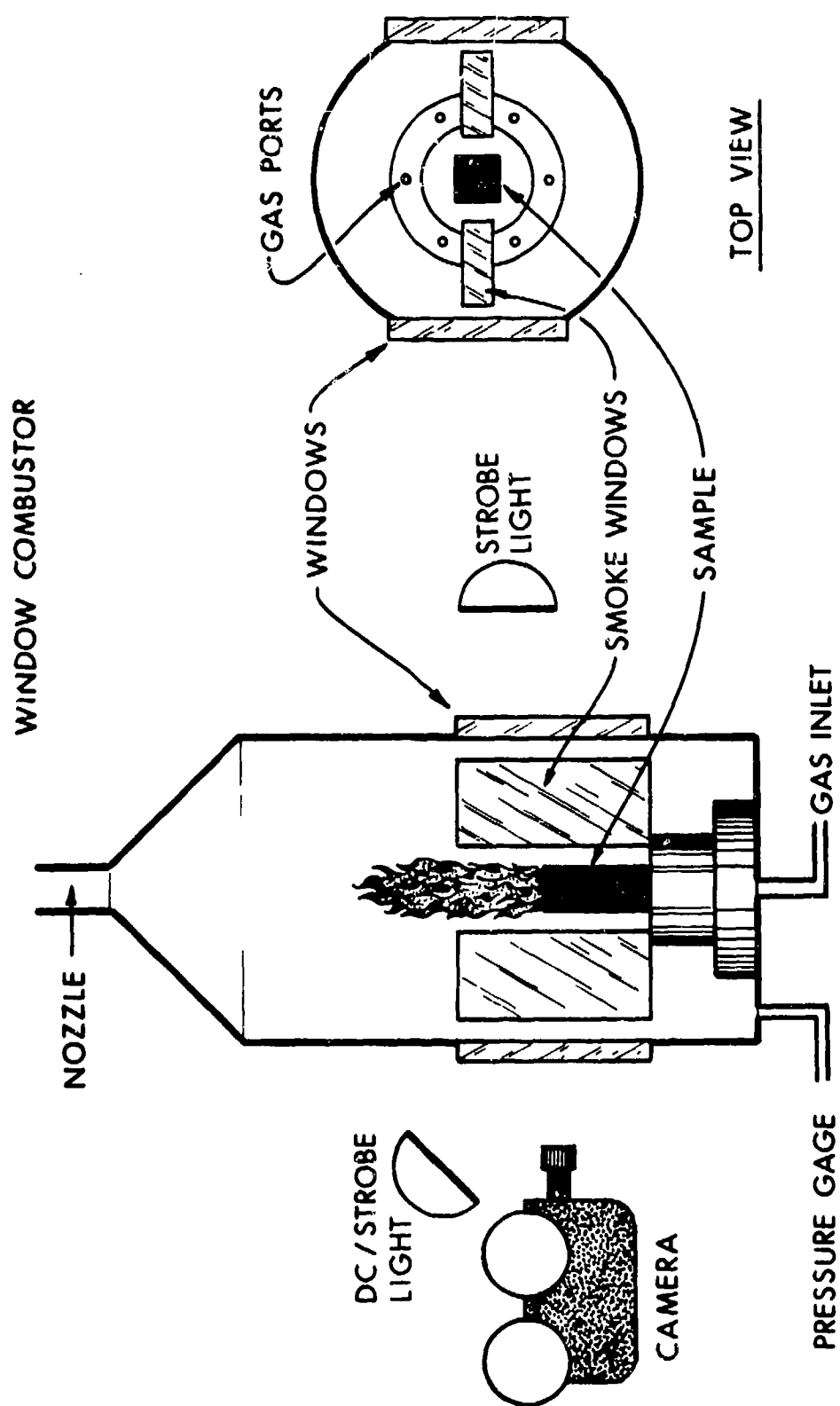


Figure 5. Schematic for Black Powder Strand Burner.

by a rapidly heated wire. A film speed of 2000 frames per second gave good time resolution and provided a time window large enough to examine the entire strand burn using 200-foot-long rolls of film. To look for deconsolidation during combustion, a 1.5- $\mu$ s pulse-width strobe light was used for back lighting. The strobe was triggered by pulses from the camera, exposing every second, third, or fourth frame chosen to meet a particular requirement. Front lighting was provided by either a 650-watt flood lamp or a second strobe light. Again, the strobes could be synchronized to provide alternate front, rear or no lighting for a consecutive sequence of frames. A piezoelectric pressure gauge monitored the chamber pressure. By measuring the pressure rise from ignition to burn-out, the burn rate of a strand of known length was measured. Burn rates were also determined from the high speed films by performing a regression analysis on the position histories of the regressing surface.

Because of the large concentration of smoke and particulate matter, it became necessary to use plastic smoke windows to limit the optical path length in the flame to approximately 8 mm. The opacity of the flame increased with pressure, and even with smoke windows, the back lighting could not be observed through the flame at pressures beyond 0.5 MPa.

Black powder strands were prepared by pressing known weights of black powder meal (passing through a 200-mesh screen) into a constant volume parallelepiped. In this manner density could be carefully controlled and adjusted as required. The meal contained 2-4% moisture and pressure was supplied by a Carver press. Typically, the black powder meal required 20 MPa to form a strand. The sides were inhibited with a thin coat of cyanoacrylate-based glue.

Examples of the high speed films are shown in Figure 6. The burn rates derived from the films are shown in Figure 7 and Table 2. Linear regression analysis of the data yields the following expression for the burn rate:

$$r = 25 p^{0.163 \pm 0.017} \quad 0.4 \text{ MPa} < p < 13 \text{ MPa.} \quad (4)$$

This equation is in good agreement with values of Belyaev<sup>16</sup> (Equation 2) and Barber<sup>21</sup> (Equation 3). The data of Belyaev shows some scatter but, for clarity, only the average curve is presented in Figure 7.

Although droplets were observed in the flame zone, there was no visual evidence for combustion-induced deconsolidation. Only in one case was there a visible suggestion that this might be occurring and this was with a sample of extremely low density (1.417 g/cm<sup>3</sup>) at a chamber pressure of 13.3 MPa. This is the lowest density of all the samples tested and the deconsolidation is not viewed as typical. The burn rate under these conditions was 51 mm/s, which is substantially larger than for any other sample.

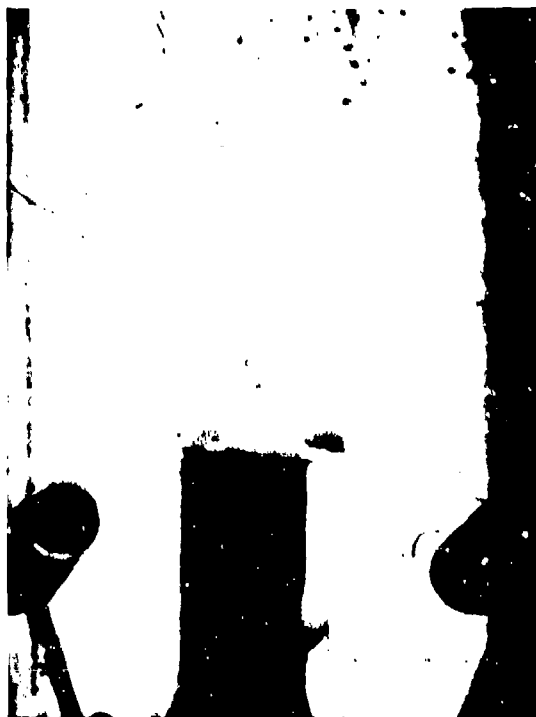
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c



b



d

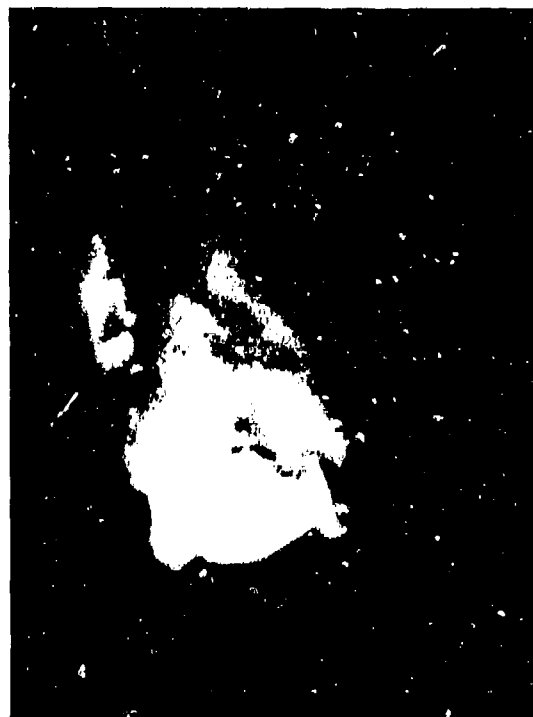


Figure 6. High Speed Photos of a Burning Strand of Black Powder.  
(a) 0.34 MPa, Front Lighting; (b) 0.34 MPa, Back  
Lighting; (c) 10 MPa, Back Lighting; (d) 10 MPa,  
Front Lighting.

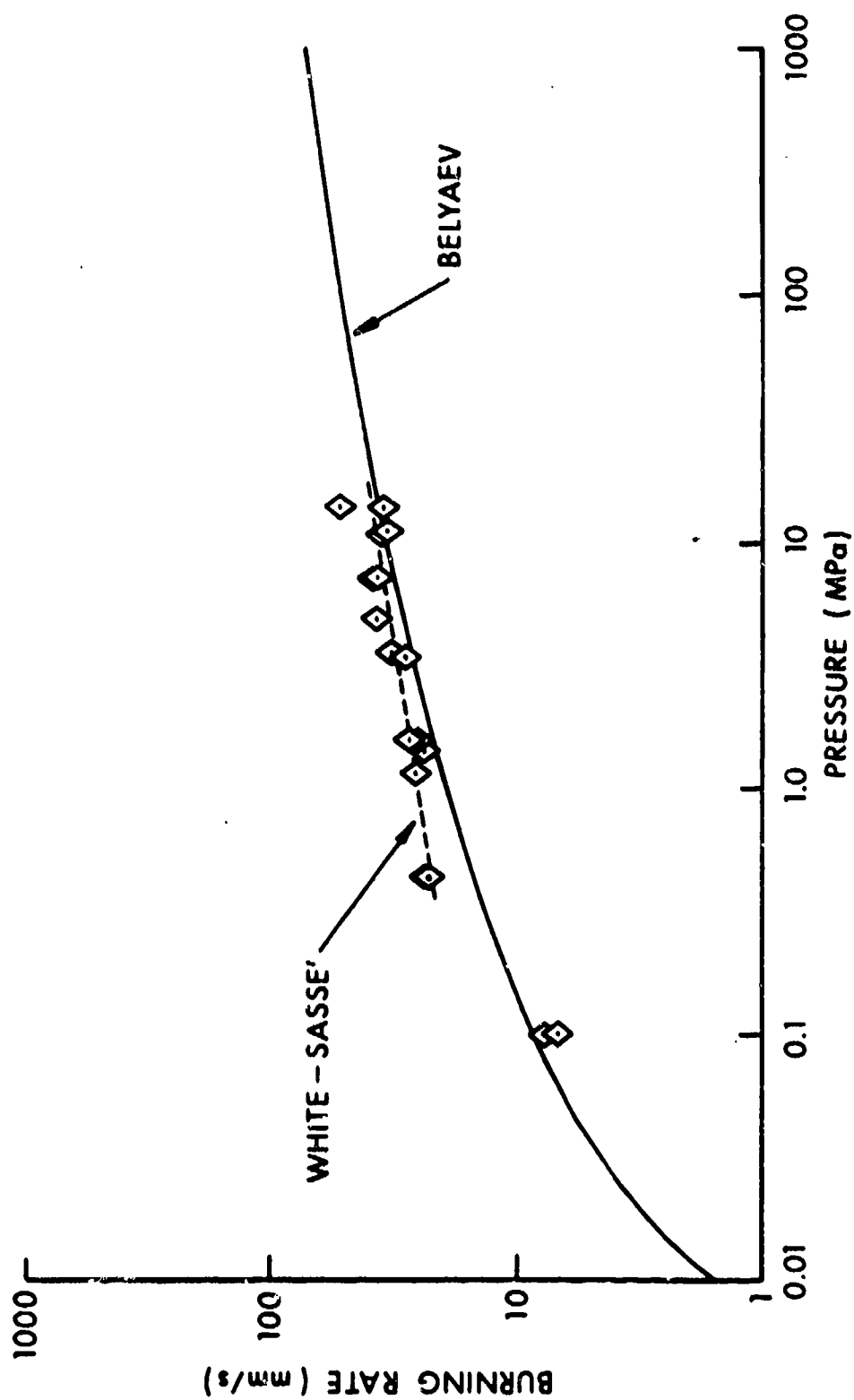


Figure 7. Comparison of Present Burn Rates with Belyaev's Data.



TABLE 2. BURN RATE DATA FOR BLACK POWDER STRANDS

<u>Pressure (gage)</u> (MPa)	<u>Density</u> (g/cm <sup>3</sup> )	<u>Burn Rate</u> (mm/s)
13.5	1.42	31.0
13.5	1.70	34.1
10.4	1.91	35.0
10.6	1.65	33.3
7.0	1.64	36.3
6.3	1.55	38.2
4.8	1.70	37.5
3.55	1.68	31.8
3.55	1.85	28.4
1.58	1.48	25.5
1.58	1.67	26.5
1.48	1.83	24.2
1.13	1.66	25.1
0.45	1.67	23.2
0.45	1.83	19.4
0.45	1.64	23.2

It can be concluded from these observations that black powder burn rate has a very weak dependence on pressure and that there is no evidence for combustion-induced deconsolidation under constant pressure. However, under conditions in which black powder is subjected to strong mechanical loading and pressure transients, such as in a vigorously ignited closed chamber or igniter tube, there is evidence that grain break-up may be taking place yielding substantially different ballistics than those predicted by the low pressure exponent burn rate.

It should be noted that the strand burner rate tests were carried out with a pre-pressurized chamber. Pressure for the closed chamber studies was provided, of course, by the combustion gases. *Could the pre-pressurized gases in the strand burner permeate the slightly porous black powder<sup>9</sup> inhibiting a porous combustion process and slowing down the burn rate when compared with the closed chamber results?* In the latter case, the combustion gases would probably not penetrate the pores of the black powder and, hence, inhibit the burning velocity. Two pieces of evidence can be presented which would negate this argument. In the first instance, the low exponent rocket motor data of Barber was taken under conditions of self pressurization. Secondly, the closed chamber results from Propellax<sup>7,9</sup> show burn rates with both low and high exponents, both under conditions of self pressurization.

#### IV. CONCLUSIONS

The general conclusions that can be drawn from this work are:

- a. there is indirect evidence of black powder grain break-up under mechanical stress and pressure transients,

b. grain break-up does not appear to be induced solely by combustion processes,

c. mechanical properties should be considered in the performance evaluation of black powder.

A number of phenomena were observed during the course of this study which were not described in the previous sections such as the presence of large droplets in the flame, a post firing residue, and burn rate dependence on both the consolidation pressure and the type of charcoal used. These effects will be described in a future report. The most important future work will be to devise an experiment for testing the mechanical properties of black powder.

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